DENITROGENATION OF DIESEL USING *IONIC LIQUID*: CHARACTERIZATION

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ABSTRACT

Diesel is widely used throughout the world on transportation mainly. But at the same time, environmental problem is increasing due to this kind of fuel. Diesel contain of several compounds like sulphur, nitrogen and so on. Nitrogen compounds for example indoles, carbazoles and pyridines can lead to health problem like coughing, shortness of breath and decreased lung function. Therefore, many researches have been done to discover how to eliminate this compound from diesel and one of them is the extraction of nitrogen compound from diesel using the ionic liquid. 3-methylimidazolium methylsulfate, ionic liquid is used in this research because of its unique properties in the extraction and synthesis process like excellent chemical and thermal stability and non-volatility for the process. This study conducts two steps of experiment. Ionic liquid is been analysed first using two method which are Fourier transform infrared spectroscopy (FTIR) and Carbon Hydrogen Nitrogen and Sulphur (CHNS) Analyser. By using Fourier transform infrared spectroscopy (FTIR), the spectrum result shows the ionic liquid has several functional groups such as alkene, aromatics and ketones according to the peaks of the graph. Based on the graph, the lowest peak is 626.46 cm-1 which can be aromatic or alkynes group. This group have C-H bond that exactly exist in the ionic liquid. While the highest peak the spectrum graph is 3126.60 cm-1 which can be carboxylic group. Meanwhile, the CHNS result shows the percentages of five major components in the ionic liquid, carbon 29.71%, hydrogen 5.46%, oxygen 33.75%, nitrogen 14.97%, and sulphur 16.11%. Those results are compared with the actual molecular formula of the ionic liquid to make sure the components are existed in the liquid. From the calculation from the CHNS analysis, the ratio of carbon is 4.9235, oxygen is 4.1988, nitrogen is 2.1274, hydrogen is 10.7826 and sulphur is 1.0000. The error is about 10% from the theoretical formula and it is acceptable.

ABSTRAK

Diesel merupakan salah satu bahan bakar yang digunakan di seluruh dunia selain daripada petrol, kerosene dan lain-lain lagi. Walaupun telah lama digunakan, diesel masih lagi mempunyai masalah yang perlu diperbaiki yang mana prestasinya masih lagi dikaji dan perlu ditingkatkan lagi supaya penggunaannya menjadi lebih efisyen. Komponen yang terdapat dalam diesel terdiri daripada sulphur, nitrogen dan lain-lain lagi. Nitrogen merupakan salah satu komponen yang masih dikaji oleh para saintis seluruh dunia yang dikira sebagai salah satu punca kualiti diesel itu sendiri menjadi tidak stabil. Nitrogen boleh menyebabkan masalah kesihatan kepada manusia seperti batuk, kanser jantung dan sebagainya. Disebabkan itu, ramai saintis sekarang ini berusaha untuk mengurangkan kadar nitrogen dalm diesel. Salah satu proses yang hangat digunakan pada masa kini ialah dengan menggunakan cecair ionik. Para saintis kini giat menjalankan eksperimen menggunakan cecair tersebut bagi mengurangkan kandungan nitrogen dalam diesel. Cecair tersebut menjadi popular kerana kestabilannya dalam proses mengurangkan kandungan nitrogen dalam diesel.

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1 INTRODUCTION

1.1 Background of study

Diesel is one of the fuels that widely used in transportation apart from petrol, Natural gas vehicle (NGV) and kerosene. Diesel contain of several compound such as sulphur, aromatic compound, nitrogen and son on. **Table 1-1** shows the composition of the components of diesel. One of them, which is nitrogen compound have many undesirable effects on diesel whether during its refining process or throughout its usage. In order to control the air pollution caused by motor vehicle emissions, a policy have been establish to adopt the most stringent requirements for the quality of engine and also the by-product caused. According to (Environment, Transport and Works Bureau, June, 2004) a diesel motor vehicle meeting the Euro IV emission standards and using diesel meeting the Euro IV specification will emit 26% less nitrogen oxides and 76% less particulates than a diesel motor vehicle meeting the Euro III emission standards and using diesel meeting the Euro IV specification.Nitrogen can be poison to the catalysts, which is harmful to its characteristics and storage stability and at the same time causing air pollution (Cheng et al., 2004). Diesel usually contains of small amount of nitrogen-based compounds which is about 0.1% to 2%. But, with the increasing boiling point of a particular diesel fraction, the nitrogen content will be increases. The classification of nitrogen compound in diesel is divided into four classes which are aliphatic amines, aniline, non-basic (five-membered pyrrolic ring) and basic (six-membered pyridinic ring systems).The extraction of nitrogen compounds from diesel is done through many methods like catalytic/noncatalytic, ion exchange, alkylation and metal-complexation. However, these method lead to the failure when the nitrogen extracted is not too much (Anantharaj et al., 2010).

Table 1-1: Result of the statistical analysis of chemical composition

Components	Values (ppmw)
Total sulphur	430.2
Total Nitrogen	271.3

1.2 Motivation and statement of problem

Demand for the clean energy nowadays is been increasing. Environmental effect is the main focus of the world that needs to be reduced while using these kinds of fuels. Therefore, in order to be flexible on using this fuels, diesel itself need to be clean and high quality so that operation properly can be done. Diesel is composed of about 271.3 ppmw of nitrogen compounds including aniline, alkyl anilines, quinoline, indole, alkyl indole and carbazole (Koriakin et al., 2010). Therefore, people come out with the denitrogenation process that can improve the quality of the diesel by removing the nitrogen compounds. Denitrogention means that, the nitrogen compound is extracted from the diesel itself. Nitrogen compounds are important in diesel but this compound seriously affects the oxidation stability of diesel. This compound is known as poisonous organic compound and not easily biodegraded (Singh et al., 2005). Nitrogen compounds are suspected of causing problems such as color and gum formation, engine deposits, and poisoning of some catalysts (Quimby, D.B., 1998). On the other hand, nitrogen dioxide is one of the by-products of diesel combustion and contributes to the formation of ground-level ozone or "smog". Ozone, O_3 is a colorless, pungent, highly reactive gas, considered as a secondary pollutant. This type of pollutant is formed by photochemical reaction driven by the action of ultraviolet light on the precursor pollutants oxides of nitrogen (NOx), and volatile organic compounds (VOCs). It also will lead to the health effects like coughing, shortness of breath and decreased lung function. They can also affect aquatic ecosystems by providing too many nutrients to aquatic plant life and then reduces dissolved oxygen levels and can ultimately harm the ecosystem (Fierro M., 2001). Therefore, ionic liquid is used as extracting agent in order to remove nitrogen compounds from diesel. Ionic liquid is become the suitable extractor agent to be used because of their excellent chemical and thermal stability. In addition they also good in solvating capability which mean they can dissolve easily in chemical.

1.3 Objectives

The following are the objectives of this research:

- To characterize ionic liquid (3-methylimidazolium methylsulfate).
- To evaluate the components of the ionic liquid (3-methylimidazolium methylsulfate).

1.4 Scope of this research

The following are the scope of this research:

i) Characterization of 3-methylimidazolium methylsulfate using FTIR analysis.

ii) Characterization of 3-methylimidazolium methylsulfate using CHNS analysis.

iii) Study the components that might be exist in the ionic liquid through the characterization process.

1.5 Main contribution of this work

This experiment main purpose is to find the right imidazalium-based ionic liquid that will remove the some percentage of nitrogen compounds from fuel. This experiment use 3-methylimidazolium methylsulfate, we will analyse the ionic liquid in order to know the components that build up that liquid. The analysis is done through by FTIR and CHNS units.

2 LITERATURE REVIEW

2.1 Overview

Due to increasingly of environmental problem related to the transportation fuels, the desulfurization and denitrogenation of fuels have become very important research topics in the production of clean automotive fuels. Diesel is one of the fuels that widely used throughout the world on transportations and industries. Therefore many researches have been done to improve the quality of this fuel. Denitrogenation of diesel using ionic liquid is one of the methods that can improve the quality of the diesel by extracting the nitrogen compound such aniline, quinoline, indole, pyridine and so on. Commonly called as ‘green solvent’, ionic liquid have been studied more because of their unique environment in the chemistry, synthesis and electrochemistry. These ionic liquids have their own interesting properties such as excellent chemical and thermal stability, non-volatility and ease of recycling. Utilizing ionic liquid is one of the goals of green chemistry because they can create cleaner and more sustainable chemistry (Niknam et al., 2009).

2.2 Physical and chemical properties of ionic liquid

Ionic liquids are the compounds that composed of ions which are in liquid form at or close to room temperature. There are several types of ionic liquid that can be categorized, which is acidic ionic liquids, basic ionic liquids and also ionic liquids containing metallic elements. The acidic ionic liquids are ones of the most important and widely used in the reaction. This ionic liquid is the one that have one or more acidic activity sites are in the framework of the ionic liquids. Compared to the acidic ionic liquids, basic ionic liquids also have one or more basic activity sites in the framework of the ionic liquids. Basic ionic liquid have the ability of providing one or more electron pairs. In addition it also has the ability of accepting one or more protons. The third type is ionic liquids containing metallic elements also known as ionic liquid crystals. Based on the metallic elements' position and the ionic liquids' state of existence, the ionic liquid crystals divided into two groups, the normal ionic liquids with an anion containing metal ion and the ionic liquids with a cation containing metal ion. Usually both of them are in the solid state. Ionic liquids become too attractive due to their chemical and physical properties (Yue et al., 2011).

The structure of ionic liquids plays a key role in so many of the interesting and useful ways especially for the synthesis in chemistry. Ionic liquid mainly composed of ions which mean anion and cation, but cation is widely used based on ammonium, imidazolium, pyridinium and so on. Cation normally has a bulk organic structure with the low symmetry. Compared to the cation, the anion ionic liquid may come in form of organic or inorganic. Figure 2-1 shows some typical structure of cation and anion of ionic liquids.

Ionic liquid also possesses unique of chemical properties for example melting point, densities, viscosities and thermal stability. Melting point of ionic liquid is the most significant property because it related with the composition and the structure itself. Melting point is determined by the selection of ionic liquids itself whether it is cation or anion. Cation ionic liquid have low melting point compared to anion because of the low symmetry structure. Therefore, cation ionic liquid is become more suitable for the synthesis in chemistry. For density part, generally ionic liquid is denser than water. Both melting point and density of ionic liquids depend on the constituent of between cation and anion (Singh et al., 2005).

By the way, the thermal decomposition depends on the nature of anion compared to cation. But, mostly the ionic liquids are stable at 400^{o}C and above that temperature. Anions have their own role because thermal decomposition inversely reacts to the increase of hydrophilicity of anion. Viscosity of ionic liquids is very important part when some chemical reaction using it as solvent media. So, several of ionic liquids are less suitable for solvent media due to their high viscosity (Singh et al., 2005).

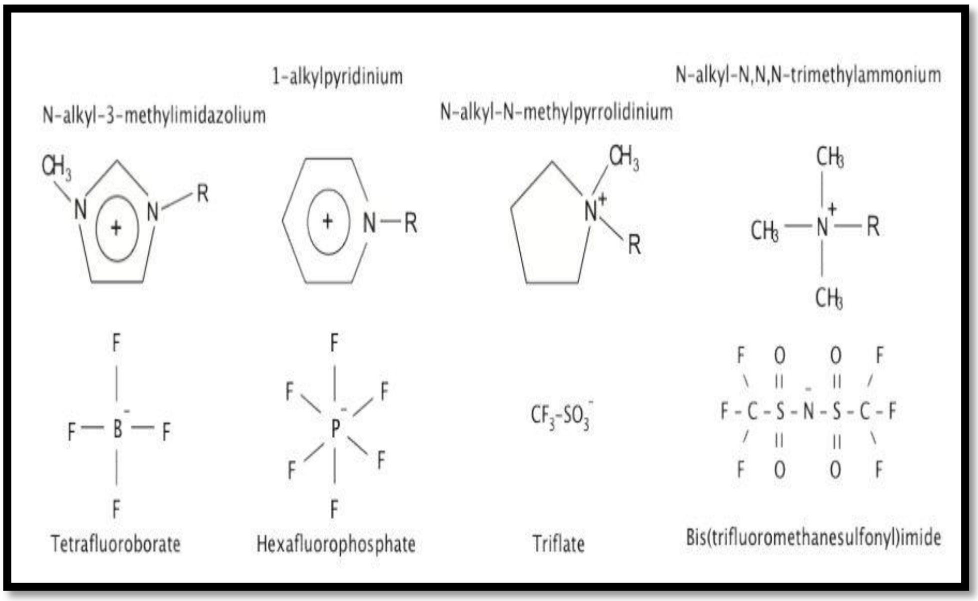

Figure 2-1: Examples of cation and anion structures typically found in ionic liquids

2.3 Review journal of removing nitrogen compound on diesel

There are several experiment have been done before regarding of removing nitrogen compound from diesel. The first one using adsorption principle, Lithium-modified mesoporous silica adsorbents (YSP-Li and MCF-Li) are used as extracting agent for nitrogen compounds in diesel. This experiment is about to study the adsorption properties of lithium (Li) modified mesoporous silica adsorbents toward nitrogen compounds in diesel. There is about 271.3 ppmw of nitrogen compounds, 430.2 ppmw of sulphur compounds and a certain level of aromatic hydrocarbons in the provided diesel used for this experiment. It takes about 48 hours for the mixture to completely settle down before it can be separated. Based on the result, at the same temperature, nitrogen compound extracted is increase when the absorbent amount is increase. Therefore, the temperature is not affect the adsorption rate of nitrogen compounds. But the readsorption of nitrogen compound is decrease after second and third trial (koriakin et al., 2010).

Besides that, nitrogen compound is extracted using two different filtering media, activated clay and silica gel in the other experiment. Based on the result, about 60% of the nitrogen compounds identified including of pyridines, quinolines and tetra-hydroquinolines were detected using gas chromatography analysis. The result shows that these two filtering media is effective by removing about 97.8 and 99.9% of the N-compounds (Mushrush et al., 2011).

An adsorptive denitrogenation of organic nitrogen compounds including pyridine, quinoline, pyrrole,and indole in diesel was conducted using three mesoporous molecular sieves (Ti-HMS, HMS, and MCM-41) in a fixed-bed adsorption. According to the result, both temperature and (liquid hourly space velocity) LHSV influence the adsorption performance on nitrogen compounds. The adsorption capacity of the adsorbent is determined by the acidity and pore structure of the adsorbent, the acid−base interaction between adsorbent and adsorbate, as well as the polarity and molecular weight of the adsorbate. By the way, the adsorption capacity is decrease within the increasing of temperature. So, the study indicate that the adsorption capacities increase in the order MCM-41 < HMS <Ti-HMS, and Ti-HMS is the most effective adsorbent for nitrogen removal (Zhang et al., 2012).

Based on one of the experiment, nitrogen compound is removed using strongly acidic ion-exchange resin (IXR), by liquid-liquid extraction. Toluene and n-dodecane is used as model oil with 99% mass purity. The experiment is to extract the basic nitrogen compound pyridine. According to the result, about 78% of nitrogen compound was extracted. But the regeneration method used for the adsorbent reduced the extracted value of nitrogen compound (Xie, L-L., et al 2010).

One of the experiments is using 3 ionic liquids which is (1-ethyl-3-methylimidazolium thiocyanate), (1,3-dimethylimidazolium methylphosphonate, and [tris-(2-hydroxyethyl)-methylammonium-methylsulfate]. These ionic liquid is using to extract one of nitrogen compound in diesel which is pyridine. The experiment is conducted on n-heptane as model oil for diesel. A simple method is using for this experiment. The mixture of both model fuel and ionic liquid is mixed and stir for 2 hours to get a good contact. The mixture then is kept for one night in order to settle down. Between the three ILs, [EMIM][SCN] shows the highest selectivity for the extraction of pyridine. This ionic liquid is selected mainly due to the immiscibility of n-heptane based on the experiment (Karolina et al., 2011).

At the same time, on the other experiment, 1-n-butyl-3-methylimidazolium bis(trifluoromethylsulfonyl)imide (BMI.NTf$_2$)] ionic liquids is used to extract the nitrogen compound from diesel. This ionic liquid is quite good in this process of denitrogenation. This experiment is using n-octane as their model oil for diesel in order to extract pyridine. Based on the experimental result, the quantity of the nitrogen compounds present in this ionic liquid phase increases due the increase of the pKa. The data shows that, this ionic liquid extracted about 80% of the pyridine in 1 wt% of n-octane (Cassol et al., 2007).

On the other part, 1-ethyl-3-methylimidazolium acetate [EMIM][OAc], 1-ethyl-3-methylimidazolium ethylsulfate [EMIM][$EtSO_4$] and 1-ethyl-3-methylimidazolium methylsulfonate [EMIM][$MeSO_3$] ionic liquids is used to extract nitrogen compound, pyridine in diesel. Based on the result, the selectivity values are higher than unity for all of the studied systems, while the distribution coefficient values are lower than unity. Therefore, these ionic liquids are suitable in order to extract the nitrogen compound but need to be in a large quantity (Shah et al., 2012).

The other one used ionic liquid 1-ethyl-3- methyl-imidazolium chloride [C_2 mim] [Cl] as extracting agent for nitrogen compound, pyridine. But in this experiment, pure dodecane is used as model oil not diesel itself. Based on the result, the percentages of pyridine that been extracted was about 70%. But due to the ratio effect and also the present of aromatic compound, the percentages reduced to 56% only. In other word, this ionic liquid is suitable extract nitrogen compound on diesel (Anugwom et al., 2011).

2.4 Summary

This chapter review the background of the ionic liquid which has been studied in this research. This chapter also review the previous work of other research that using ionic liquid to remove nitrogen compound from diesel and its capability.

3 MATERIALS AND METHODS

3.1 Overview

This paper presents the methodology of the experiment that will be done in this research. There are two experiments that will be conducted which are analysis of ionic liquid (3-methylimidazolium methylsulfate) using FTRIR and CHNS method.

3.2 Chemicals

Ionic liquid, 3-methylimidazolium methylsulfate is obtained from Universiti Teknologi PETRONAS (UTP).

a) ***Ionic liquid (3-methylimidazolium methylsulfate)***

Ionic liquid use for this experiment was 3-methylimidazolium methylsulfate. It is corrosive to skin, eyes, and respiratory system. Its liquid or spray mist may produce tissue damage, particularly in mucous membranes of the eyes, mouth and respiratory tract. If contact to the skin may produce burns. If contact to the eyes can result in corneal damage or blindness. Inhalation of the spray mist may produce severe irritation of respiratory tract, characterized by coughing, choking, or shortness of breath. It is a corrosive material that may cause serious injury if ingested. If contacts to the eyes occur, remove any contact lenses. Flush eyes with running water for a minimum of 15 minutes, occasionally lifting the upper and lower eyelids. If the chemical gets spilled on a clothed portion of the body, remove the contaminated clothes as quickly as possible and protecting your own hands and body. Place the victim under a deluge shower. If the chemical touches the victim's exposed skin, such as the hands: Gently and thoroughly wash the contaminated skin with running water and non-abrasive soap.

3.3 Methodology

a) Characterization of ionic liquid using Fourier Transforms Infrared Spectroscopy (FTIR) Analysis

FTIR equipment is setup first before the analysis can be done. GS10683-2 - GE flat crystal plate is placed in the unit. The plate is approached with the needle to get the standard background spectrum. The standard spectrum will be compared with the sample spectrum later. The sample which is ionic liquid is dropped on the GE plate and then analysis is run. After the analysis is done with the spectrum result, the GE plate is clean with acetonitrile. The procedure is repeated until the spectrum result is consistent. **Figure 4.1** shows the spectrum graph for the analysis of the ionic liquid. The analysis of the wavelength data can be done by refer table of IR Absorptions for Representative Functional Groups in the appendix. The functional group and molecular motion were found based on the table of IR Absorptions for Representative Functional Groups.

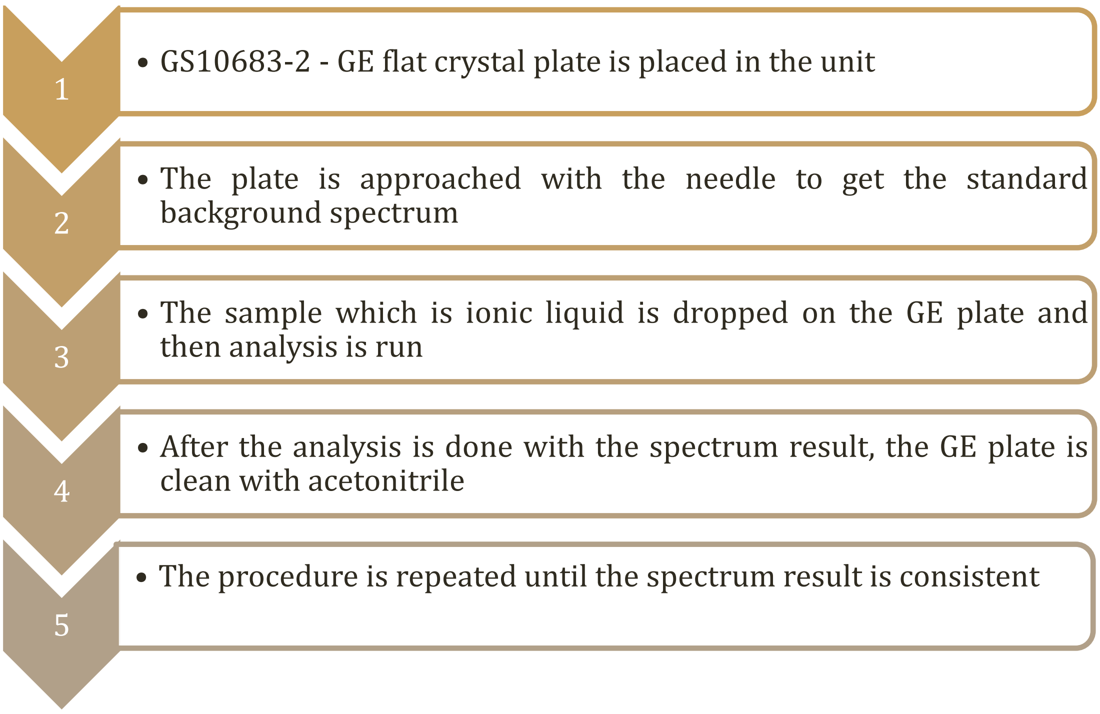

Figure 3-1: Flowchart of the FTIR analysis

b) *Characterization of ionic liquid using CHNS Analysis*

Ionic liquid is analysed to see how much amount of carbon, hydrogen, nitrogen, sulphur and oxygen exist in the sample. After been prepared in the vial about 2 ml, the sample is send to the central. This process is been done in the central laboratory by the laboratory instructor.

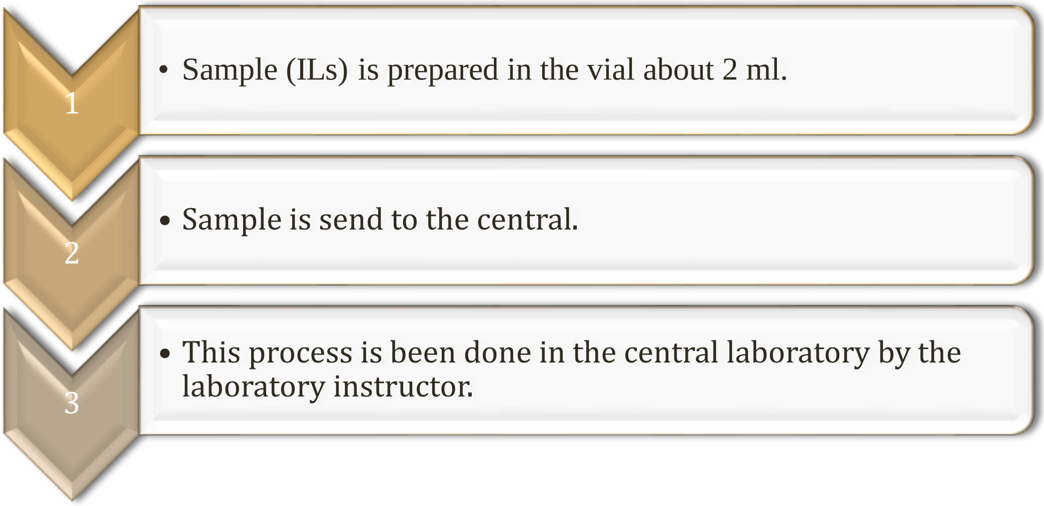

Figure 3-2: Flowchart of the CHNS analysis

Figure 3-3: Sample of ionic liquid

4 CHARACTERIZATION OF *IONIC LIQUID*

4.1 Overview

This chapter review about the results of this thesis after the experiments is done. There are two results that will review here which are FTIR analysis and CHNS analysis. Both FTIR and CHNS are all about the characterization of the ionic liquid itself.

4.2 Results of FTIR Spectroscopy Analysis

The molecular formula for the ionic liquid, 3-methylimidazolium methylsulfate is C5H11N2SO4. **Figure 4.2** shows the structural formula of the sample. Therefore the analysis from the FTIR and CHNS will be compared with the molecular formula theory. Based on the wavelength result from the FTIR analysis, there are several functional group may exist in the ionic liquid, 3-methylimidazolium methylsulfate. Based on **Figure 4.1** which is the result of FTIR analysis, **Table 4.1** shows the functional group that can be concluded in the ionic liquid according the peak of the graph. The lowest peak shows in the graph is 626.46 cm-1 which can be aromatic or alkynes group. This group have C-H bond that exactly exist in the ionic liquid. While the highest peak the spectrum graph is 3126.60 cm-1 which can be carboxylic group. This group consist of O-H stretch which also has been recognized in the sample. The other group is amine which consists of N-H bond. This is same with the structural formula of the ionic liquid for the imidazolium part.

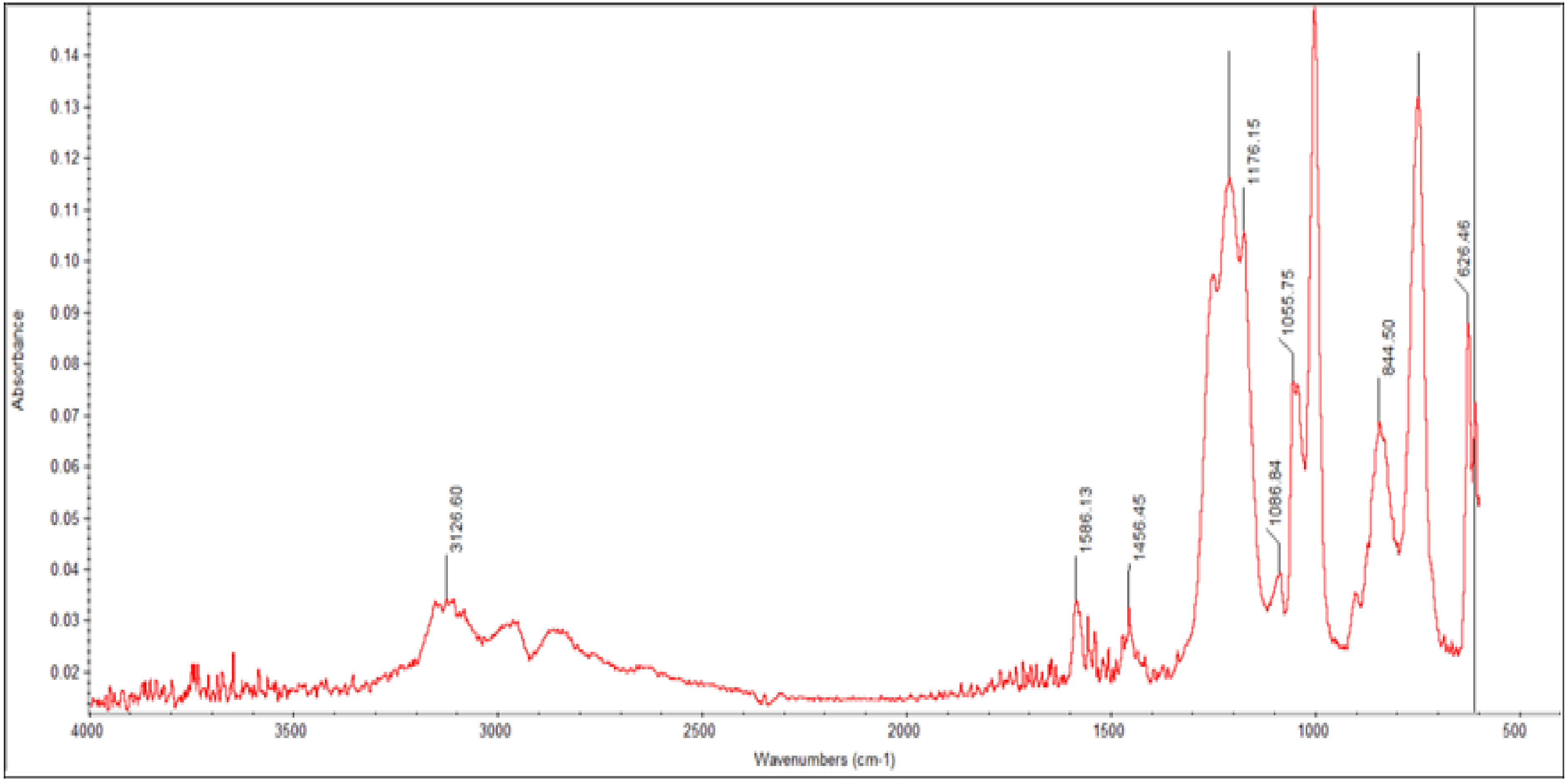

Figure 4-1: FTIR analysis result

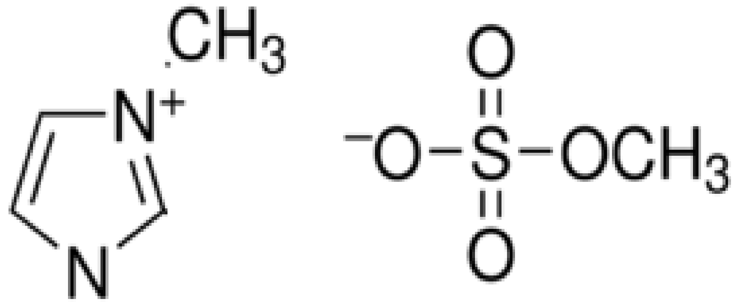

Figure 4-2: Structural formula of the ionic liquid

Table 4-1: Functional group of the components

Wavelength (cm^{-1})	**Functional Group**	**Molecular Motion**
626.46	1. aromatics 2. alkynes	1. C-H bend (meta) ~690 2. acetylenic C-H bend 650-600
844.50	1. alkenes	1. C-H bend (disubstituted -1,1) ~890
1055.75	1. ketones 2. amines	1. C-C stretch 1300-1100 2. C-N stretch (alkyl) 1200-1025
1086.84	1. ketones 2. amines	1. C-C stretch 1300-1100 2. C-N stretch (alkyl) 1200-1025
1176.15	1. alcohols 2. ketones	1. C-O stretch 1260-1000 2. C-C stretch 1300-1100
1456.45	1. alkenes	1. CH2 bend ~1465
1586.13	1. amines	1. N-H bend 1640-1500
3126.60	1. carboxylic acid	1. O-H stretch 3400-2400

4.3 Results of CHNS Analysis

While for CHNS analysis, the result of the percentages for the components in the ionic liquid which are carbon, hydrogen, sulphur, nitrogen and oxygen is used to compare with the actual molecular formula of the ionic liquid. **Table 4.2** shows the comparison of the components both from actual and from the CHNS analysis. Based on CHNS analysis, the sample consists of 29.71% of carbon, 5.463% of hydrogen, 14.97% of nitrogen, 16.107% of sulphur and 33.75% of oxygen. There are no big different in both value from actual molecular formula and CHNS analysis. Oxygen is the major component in the sample followed by carbon, sulphur, nitrogen and the minor part is hydrogen. The comparisons are calculated by dividing the value of components from the CHNS analysis with their own molecular weight. Then, using the smallest value from the calculation, the other values are divided to get the ratio. Therefore, from this calculation, the comparison is been made as shown in **Table 4.2**.

Table 4-2: Comparison CHNS and actual molecular formula

ELEMENTS	CHNS (%)	MOL.WEIGHT	CHNS/MW	RATIO
C	29.71	12.0107	2.4736	4.9235
O	33.75	15.9994	2.1095	4.1988
N	14.97	14.0067	1.0688	2.1274
H	5.46	1.0079	5.4172	10.7826
S	16.11	32.0650	0.5024	1.0000

The comparison of the components composition with the calculated values should indicate identity and purity of the synthesized compound. **Table 4.3** shows the error between the calculation from CHNS analysis and actual molecular formula. If the differences are less than ±10% of the actual values, in case of possible contamination like water and so on.

Table 4-3: Comparison CHNS and actual molecular formula: Error

ELEMENTS	ACTUAL	RATIO	ERROR (±%)
C	5	4.9235	1.53
O	4	4.1988	-4.97
N	2	2.1274	-6.37
H	11	10.7826	2.17
S	1	1.0000	0

5 CONCLUSION

5.1 Conclusion

This project focuses about the characterization of ionic liquid, 3-methylimidazolium methylsulfate, in order to remove nitrogen compound from diesel. Ionic liquid also play an important role in this project. Therefore, by characterization we will know whether it is suitable or not for the process. By using the suitable one, the maximum quantity of nitrogen compound can be extracted from diesel.

5.2 Future Work

Therefore, based on this study, denitrogenation of diesel using ionic liquid can be done in the future. The performance of this ionic liquid can be tested whether it can extract nitrogen compound in diesel such as pyridine, indole and so on. The extraction process is quite simple which mean just mix the ionic liquid with the model oil like n-dodecane before we can continue further with diesel itself in order to make sure the ionic liquid is suitable.

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